# SYNTHESIS AND CHARACTERIZATION OF SILVER NANOPARTICLES FILLED EPOXY COMPOSITE

NURUL AKMA BINTI REDUAN

Master of Engineering (Chemical)

UNIVERSITI MALAYSIA PAHANG

## SUPERVISOR'S DECLARATION

We hereby declare that, we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Engineering (Chemical).

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## STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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JULY 2018

## ACKNOWLEDGEMENTS

First and above all, I praise God, the Almighty for providing me this opportunity and granting me the capability to proceed my study successfully. I am thankful to my honourable supervisors, Dr. Suriati Binti Ghazali and co-supervisor, Dr. Saidatul Shima Binti Jamari, for their guidance, support and encouragement throughout the course of my graduate studies. I have learned so much from them in so many ways, especially how to be independence scientist with out of box thinking. Their guidance during the course of this work allowed me to grow as a scientist, a teacher and overall sensible human being.

I would like to acknowledge Ministry of Higher Education and Universiti Malaysia Pahang (UMP), Malaysia for funding of this work under grant number of RDU121413 and GRS1503147. In this regards, I would like to thank to Associate Professor. Dr. Cheng Chin Kui, Deputy Dean of Research and Post Graduate Studies, Assoc. Prof. Mohammad Dalour Hossen Beg for his valuable suggestion during my proposal defense and Dr Mohd Bijarimi Bin Mat Piah for his advice and knowledge. I would like to extend my gratitude to the technical staff, lab technician, faculty staff and members of Chemical and Natural Resources Engineering during my postgraduate studies, I enjoyed working with you and learning from all of you.

I wish to express my sincere thanks to my husband, Airoddin bin Paiman, who has loved me unconditionally, advice and unconditional support. Through every hardship or accomplishment, he has been behind me every step of the way and this work is a direct result of that. I know that you have made a lot of sacrifice to help me to achieve my goals and I am praying that the Almighty will repay your sacrifice back. Very special thanks to my mother, Faridah binti Yusop for unconditionally support and encourage me to further study at this level. Lots of thank to my entire family members and their love and unwavering support has always been the bedrock upon which every worthwhile achievement in my life has been built.

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## LIST OF SYMBOLS

| | |
|---|---|
| α | Alpha |
| Å | Angstrom |
| cm | centimetre |
| Cu | Copper |
| ° | Degree |
| °C | Degree Celsius |
| δ | Delta |
| C=C | Double bond |
| Fe | Ferum |
| $T_g$ | Glass Transition |
| g | Gram |
| Au | Gold |
| Hz | Hertz |
| > | Is greater than |
| < | Is less than |
| kV | Kilo Volt |
| λ | Lambda |
| E'' | Loss Modulus |
| Mpa | Mega Pascal |
| μ | Micro |
| mL | Mili-Litre |
| mM | Mili-Molar |
| x | Multiple or Times |
| nm | Nanometre |
| - | Negative or negative charge |
| N | Newton |
| Ni | Nikel |
| $N_2$ | Nitrogen |
| $cm^{-1}$ | One per centimetre |
| Pd | Palladium |
| ( ) | Parentheses |
| % | Per cent |
| Pt | Platinum |
| . | Point |

| | |
|---|---|
| + | Positive or positive charge |
| Q | Power Unit |
| : | Ratio or colon |
| I, II,..i,.. | Roman Numeric System |
| Ag | Silver |
| C-C | Single bond |
| / | Slash |
| E’ | Storage Modulus |
| ϴ | Theta |
| $ | US dollar |
| X1 | Variable 1 |
| x2 | Variable 2 |
| V | Volt |
| W/g | Watt per gram ( Heat Flow) |
| 0 | Zero electron |
| Zn | Zinc |
| ZnO | Zinc Oxide |

# LIST OF ABBREVIATIONS

| | |
|---|---|
| $BH_4^-$ | Anion Borohydride |
| CTAB | Cetyltrimethylammonium bromide |
| CTAC | Cetyltrimethylammonium chloride |
| $Cl^-$ | Chloride ion |
| DTG | Derivative thermal gravimetric analysis |
| DSC | Differential scanning calorimeter |
| 1D | 1 dimensional |
| 2D | 2 dimensional |
| 3D | 3 dimensional |
| DTAB | Dodecyltrimethylammonium |
| DMA | Dynamic mechanical analysis |
| FCC | Face center cubic |
| FESEM | Field emission scanning electron microscopy |
| FTIR | Fourier transform infrared |
| $H_2$ | Hydrogen gas |
| OH | Hydroxide |
| ICPMS | Inductive coupled plasma mass spectroscopy |
| IPA | Isopropanol |
| $Ag^0$ | Metallic silver |
| $T_m$ | Melting temperature |
| PVA | Poly-vinyl alcohol |
| PVP | Poly(vinylpyrrolidinone) |
| pH | Potential of hydrogen |
| PCB | Printed circuit board |
| rpm | Revolutions per minutes |
| SEM | Scanning electron microscopy |
| AgNPs | Silver nanoparticles |
| $NaBH_4$ | Sodium borohydride |
| NaCl | Sodium chloride |
| $Ag^+$ | Silver ion |
| $AgNO_3$ | Silver nitrate |
| SPR | Surface plasmon resonance |
| $T_{5,10,50}$ | Temperature degradation at 5, 10 and 50 per cent of weight loss |
| TGA | Thermal gravimetric analysis |

| | |
|---|---|
| TEM | Transmission electron microscopy |
| UV-Vis | Ultraviolet-visible absorptions spectroscopy |
| XRD | X-ray diffraction analysis |

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## ABSTRAK

Dalam kajian ini, nanopartikel perak (AgNPs) telah berjaya disintesis melalui kaedah kimia iaitu tindak balas redox. Argentum Nitrate, $AgNO_3$ telah digunakan sebagai prekursor, Natrium Borohydride ($NaBH_4$) sebagai agen penurunan dan Cetyltrimethylammonium Bromide (CTAB) sebagai surfaktan. Kesan terhadap tindak balas suhu, kepekatan $AgNO_3$ dan NaCl telah disiasat untuk mensintesis AgNPs dengan bentuk yang seragam dan taburan partikel yang sekata. AgNPs yang telah disintesis dicirikan dengan UV-VIS, TEM, XRD, dan FESEM, manakala, saiz purata diukur menggunakan perisian Imej-J. Puncak belauan XRD mengesahkan bahawa sampel yang disediakan adalah AgNPs berdasarkan kedudukan puncak belauan pada 38.4° dan 44.8°. Parameter sintesis pada suhu 60°C, 1.00 mM dan 3.0 mM kepekatan $AgNO_3$ dan NaCl masing-masing menunjukkan keadaan optimum dengan taburan partikel yang seragam, tanpa penggumpalan dan mempunyai bentuk sfera yang terbentuk seragam pada akhir tindak balas. Penambahan NaCl juga mengubah keadaan tindak balas larutan iaitu pH. Kajian ini menunjukkan larutan pada pH 7.5 dapat menghasilkan pembentukan partikel yang lebih baik dan tanpa penggumpalan partikel terbentuk. Sementara itu, kajian kestabilan partikel melalui analisis UV-Vis dan TEM menunjukkan bahawa AgNPs yang disintesis kekal stabil sehingga satu bulan. Pelaksanaan pemindahan dari fasa akueus ke fasa organik dalam kajian ini bertujuan meningkatkan taburan partikel yang seragam dalam sistem polimer epoksi (dalam penyediaan komposit). Morfologi analisis FESEM menunjukkan partikel AgNPs disebarkan dengan baik dalam sistem epoksi. Dalam kajian ini, komposit-komposit terisi AgNPs dikaji sebagai kesan kandungan partikel dalam peratusan volum (vol %) terhadap sifat-sifat terma dan terma-mekanikal. Penambahan AgNP dalam epoksi polimer adalah bertujuan untuk meningkatkan sifat terma dan terma-mekanikal komposit yang dihasilkan. Sifat terma dan terma-mekanikal dari analisis TGA, DSC, dan DMA menunjukkan bahawa komposit epoksi yang terisi AgNPs mempunyai ciri-ciri haba terma-mekanikal yang lebih tinggi berbanding polimer epoksi yang tidak terisi. Analisis TGA menunjukkan sampel AgNPs yang dimuatkan pada 1.0 vol% direkodkan sebagai nilai tertinggi sifat terma berbanding dengan komposit AgNP yang tidak terisi. Melalui analisis DMA, sampel pada 0.2 vol% terisi AgNPs menunjukkan sifat-sifat terma-mekanikal komposit epoksi terisi AgNPs yang lebih tinggi dengan peningkatan 47-56% dalam modulus dan delta tan berbanding dengan komposit epoksi AgNP yang tidak terisi. Berdasarkan hasil ujikaji, sifat-sifat terma dan terma-mekanikal dapat dinyatakan bahawa komposit epoksi terisi AgNPs boleh digunakan sama ada dalam bidang kejuruteraan atau dalam pelbagai aplikasi.

## ABSTRACT

In this study, silver nanoparticles (AgNPs) were successfully synthesized via chemical reduction method. Silver nitrate ($AgNO_3$), was used as a precursor, Sodium Borohydride ($NaBH_4$) as a reducing agent and Cetyltrimethylammonium Bromide (CTAB) as a surfactant. The effect of reaction temperature, concentration of $AgNO_3$ and NaCl were investigated in order to synthesize AgNPs with uniform shape and narrow particles distribution. The synthesized AgNPs was characterized by UV-VIS, TEM, XRD, and FESEM while; the average size was measured by Image-J software. XRD diffraction peak confirm that the samples prepared are AgNPs based on the peak position at 38.4° and 44.8°. Synthesis parameter at 60°C, 1.00 mM and 3.0 mM of reaction temperature, concentration of $AgNO_3$ and NaCl respectively shows an optimum condition which produced AgNPs with narrow particle distribution, no agglomeration and uniform spherical shape at the end of reaction. The addition of NaCl affected the state of the reaction solution which is pH. This study shows solution at pH 7.5 gave better particles formation with no appearance of agglomerated particles. Meanwhile, stability study by UV-Vis and TEM analysis shows that the as-synthesized AgNPs was remain stable up to 1 month. The implementation of aqueous to organic phase transfer technique in this study is aimed to improve particles dispersivity in the epoxy polymeric system (composite preparation). FESEM analysis showed the AgNPs were dispersed well in the epoxy system. The composites of epoxy and AgNPs composite were fabricated as a function of particles concentration in the volume percentage (vol %) on thermal and thermo-mechanical properties of composite. Incorporation of AgNPs in the epoxy system is aimed to improve the thermal and thermo-mechanical properties of the resultant composite. The thermal and thermo-mechanical properties from TGA, DSC and DMA analysis shows that AgNPs- filled epoxy composite have higher thermal and thermo-mechanical properties compared to unfilled epoxy polymer. TGA analysis showed that, sample at 1.0 vol % AgNPs was recorded in the highest value of thermal properties compared to the unfilled AgNPs. From DMA analysis, samples at 0.2 vol% shows the thermo-mechanical properties of AgNPs-filled epoxy composite obtained 47-56% increment in the storage modulus and tan delta compared to unfilled AgNPs epoxy composite. Based on experimental results, thermal and thermo-mechanical can be conclude that AgNPs-filled epoxy composite can be used either in engineering or in various applications.

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